X-ray diffraction measurement of doping induced lattice mismatch in *n*-type 4H-SiC epilayers grown on *p*-type substrates

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High-resolution x-ray diffractometry was used to measure the lattice mismatch and misorientation in *n*-type 4H-SiC epilayers grown homoepitaxially on *p*-type 4H-SiC as function of different nitrogen doping levels. The spatially averaged lattice mismatch increased from 1.0×10^{-5} to 4.0×10^{-5}, 6.3×10^{-5}, 8.8×10^{-5}, and 11.6×10^{-5} in epilayers doped 4.1×10^{17} cm^{-3}, 2.6×10^{18} cm^{-3}, 1.7×10^{19} cm^{-3}, 2.2×10^{19}, and 4×10^{19}, respectively. The resolved multiple subsidiary peaks in the rocking curve of the epilayers doped 2.2×10^{19} and 4×10^{19} cm^{-3} are likely due to high density of domain boundaries. The increase in mismatch with doping, is attributed to the substitutional nitrogen incorporated preferentially in the host carbon sites of the 4H-SiC epilayer. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1606497]

High-resolution x-ray diffraction (HRXRD) and reciprocal space mapping (RSM) were utilized to investigate the presence of lattice mismatch and misorientation in five 4H-SiC epilayers having different nitrogen doping concentration. The *n*-type epilayers were 2 μm thick, with nitrogen doping concentrations (as verified by secondary ion microscopy) of 4.1×10^{17} (71A′), 2.6×10^{18} (66A), 1.7×10^{19} (12B), 2.2×10^{19} (75B), and 4×10^{19} cm^{-3} (79B), grown homoepitaxially on the Si face of aluminum-doped, *p*-type substrates with resistivities of 10.8, 9.49, 4.1, 4.2, 9.9 Ω cm, respectively, and tilted at approximately 8° off the (0001) basal plane. All the substrates and epilayers were produced by Cree, Inc.[1] and the samples used were sawed into 5 mm squares.

The analysis was mainly carried out on a Bede D1[2] triple-axis diffractometer equipped with a four-reflection asymmetric channel-cut beam conditioner selecting the Cu $K\alpha_1$ line only and a two-reflection asymmetric analyzer (Si 220 reflection). Depth resolved analyses by means of asymmetric reflections were also performed in a triple-axis diffractometer equipped with a Ge-channel cut beam conditioner and a Ge (111) analyzer. In addition to the common $000\bar{4}$ reflection, a total of three asymmetric reflections, $01\bar{1}6$, $01\bar{1}7$, and $01\bar{1}9$ were initially used to investigate depth dependent variations of the epilayers.

For all the samples (except 71A′ with lowest epilayer doping), two well-shaped peaks are generally present in the double-axis rocking curves (RCs) (see insets of Fig. 1). A natural assumption here is that the separation of the two peaks would be solely caused by the lattice mismatch between the epilayer and substrate, derived from[3]

$$\Delta d/d_0 = -\Delta\omega \cot\theta_B, \tag{1}$$

where Δd is the change in lattice constant, and d_0 is the reference lattice constant (0.252 nm for 0004 reflection)[4] of 4H-SiC. However, Eq. (1) is valid only when there is negligible misorientation between the (0001) lattice planes of the epilayer and substrate. A remarkable feature of the measured highly doped 4H-SiC epilayers is that they are generally tilted against the substrate by small tilt angles. Such misorientations can also cause peak splitting. By rotating the crystal by 180° along the crystal surface normal to measure another RC, one can exclude the contribution from lattice tilts in cases of low tilt angles.[3] However, this diffraction scheme has several drawbacks, such as the difficutly in determining the peak positions when the peaks are wide and heavily overlapped. Therefore, a more precise and delicate RSM method for measuring the lattice mismatch and misorientation simultaneously was employed.

Subsequent HRXRD analyses on the samples were restricted to 0004 reflections to record a set of double-axis RCs and triple-axis RSMs per sample. Figures 1(a)–1(d) are the RSMs measured from samples 66A, 12AB, 75B, and 79B, respectively. The corresponding RCs (measured without the analyzer) are plotted in the insets. The full width at half maximum (FWHM) value of each diffraction peak is typically in the range 20–30 arc sec with an open incident beam (about 1 mm×5 mm), indicating high crystalline quality of the substrates and epilayers.

The RSMs in Fig. 1 can be understood from the schematic diagram of Fig. 2. A reciprocal lattice vector (RLV), $\mathbf{h}_0$, is defined as a vector that is perpendicular to the diffracting planes [here substrate (0004) planes] with magnitude $|\mathbf{h}_0| = 1/d_0$, where d_0 is the spacing of the diffraction planes. If an epilayer with spacing d is tilted against the substrate by a small angle $\Delta\theta$ [Fig. 2(a)], the corresponding RLV, $\mathbf{h}$, which is perpendicular to the diffracting planes of the epilayer, is apparently inclined against $\mathbf{h}_0$ by $\Delta\theta$ with $|\mathbf{h}| = 1/d$. In reciprocal space, the difference between the two RLVs is then represented by $\Delta Q_z = \cos\Delta\theta/d - 1/d_0 \approx -\Delta d/d_0^2$ and

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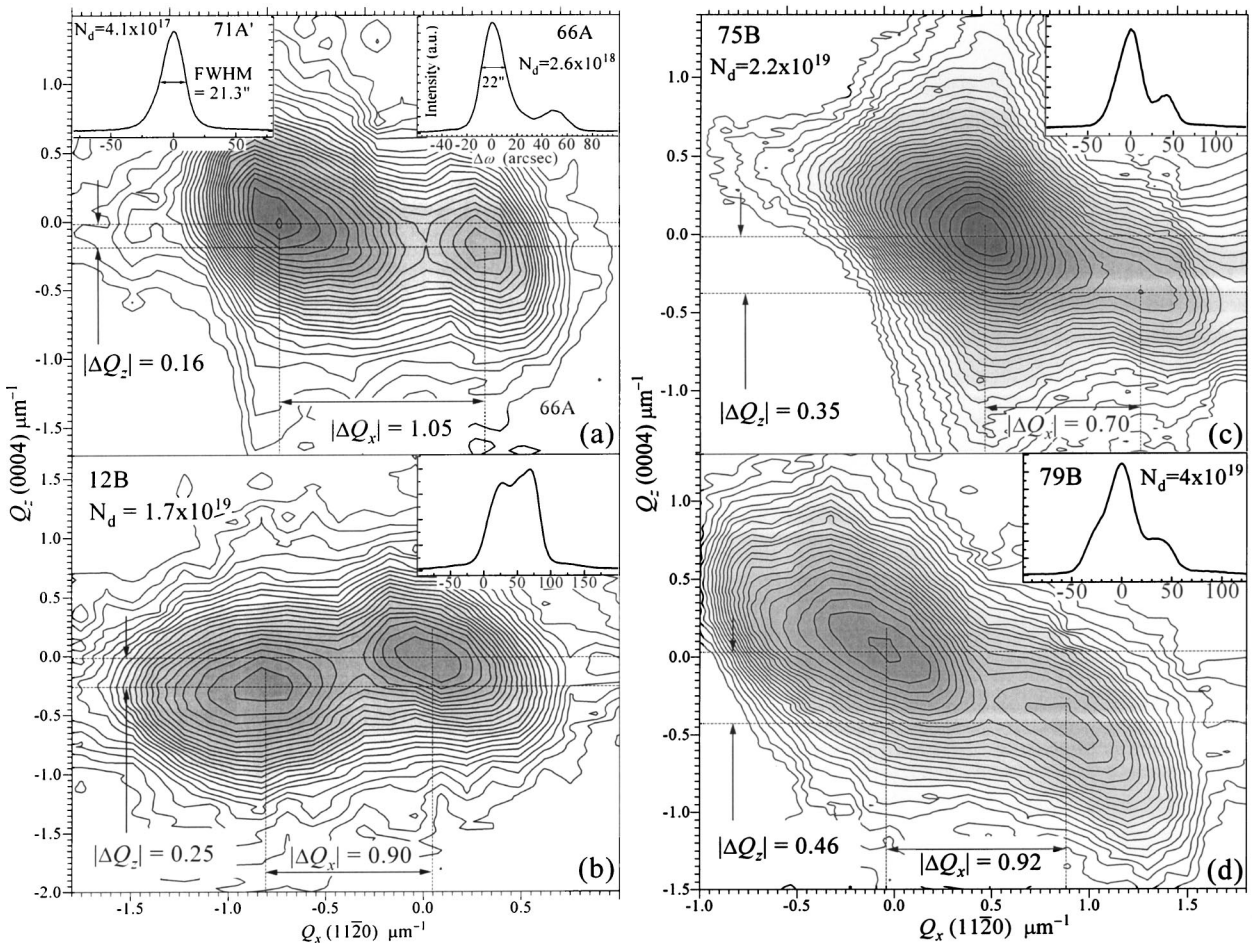

FIG. 1. HRXRD RSMs of samples (a) 66A, (b) 12B, (c) 75B, and (d) 79B. 0004 reflection, Cu $K\alpha_1$ radiation. Insets show the corresponding double-axis RCs. The upper-left inset of (a) is the double-axis RC of sample 71A.

$\Delta Q_x = \sin\Delta\theta/d \approx \Delta\theta/d_0$ along the perpendicular and parallel directions with respect to the diffracting planes of the substrate, respectively [Fig. 2(b)]. Here the approximations are well justified when $\Delta\theta$ and $\Delta d = d - d_0$ are both very small. From the ΔQ_z and ΔQ_x values measured from the RSM, one can consequently derive the lattice mismatch and lattice misorientation, respectively, as

$$\Delta d/d_0 = -\Delta Q_z d_0,$$
$$\Delta\theta = \Delta Q_x d_0. \tag{2}$$

For 4H-SiC 0004 reflection, $d_0 = c/4$, where $c = 1.008$ nm is the lattice unit constant of the substrate along [0001].[4] Based on the above principle, the $\Delta d/d_0$ values are 4.0, 6.3, 8.8, and 11.6×10^{-5} for Figs. 1(a), 1(b), 1(c), and 1(d), respectively. Meanwhile, the four corresponding $\Delta\theta$ values are 54.6, 46.8, 36.4, and 47.8 arc sec. Comparing the lattice tilt angles with the peak separations in the RCs of Fig. 1, one can see that the peak splitting is mainly caused by the lattice tilt.

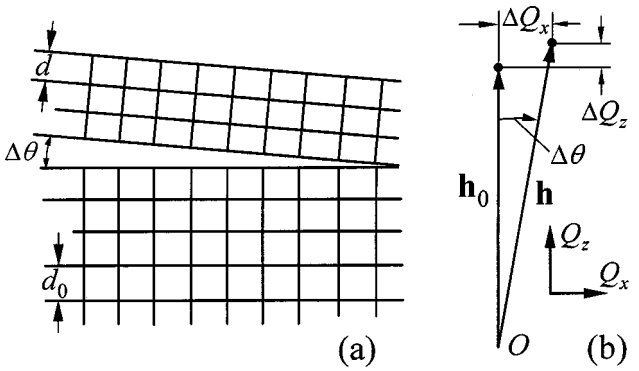

FIG. 2. Schematic representation of the epilayer tilted against the substrate. (a) Real space. (b) Reciprocal space.

Figure 3 is a SWBXT backreflection image of sample 75B (2.2×10^{19} cm^{-3}), where the misorientation of the epilayer causes its image to shift along the horizontal [11$\bar{2}$0] direction (perpendicular to the projection of the diffraction vector g on the topograph). Due to the image shift, the topograph is slightly blurred, but apparently the sample is overall of high crystalline quality, containing no obvious subgrain boundaries in the central region. This directly confirms that the double peaks in the RCs and RSMs are not caused by defects in the substrates, but by the slight differences in lattice constant and orientation between the epilayers and substrates.

As shown in the upper-left inset of Fig. 1(a), the RC measurement shows that the lowest doped sample 71A′ has no noticeable peak splitting. The FWHM of the 0004 RC is approximately 21 arc sec, and the intensity profile is reasonably invariant to the rotation around the surface normal and also invariant to the translation of the sample, which implies high crystalline quality of the epilayer across the sample. The change in lattice parameter and orientation of the sample, if any, is therefore not resolvable by these measurements. How-

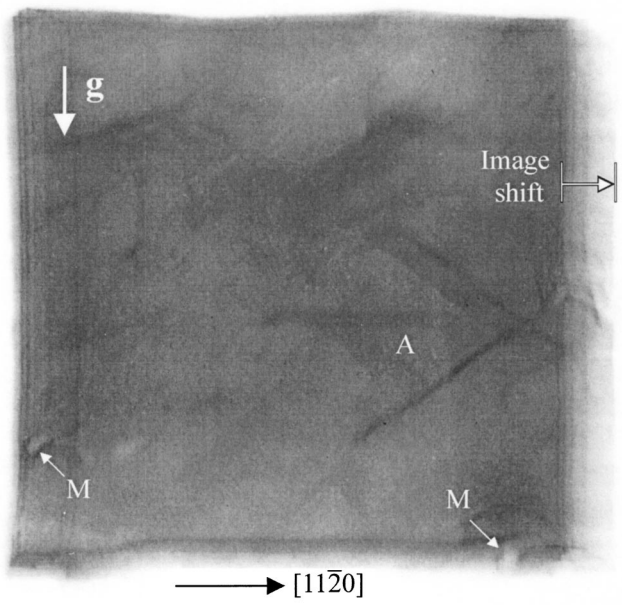

FIG. 3. SWBXT back reflection image of sample 75B ($N_d = 2.2 \times 10^{19}$ cm^{-3}) showing the image shift along the horizontal [11$\bar{2}$0] direction (perpendicular to the projection of the diffraction vector g on the topograph) of the epilayer caused by the misorientations. Field width 0.5 mm. 00$\bar{1}$6 reflection (and harmonics). *M*: micropipes. *A*: Arrow inscribed at the backside indicating the [11$\bar{2}$0] orientation.

ever, for other samples with higher dopant concentrations, clear peak splitting with at least two local maxima are always observed. The RCs of the heaviest doped samples 75B and 79B (2.2 and 4×10^{19} cm^{-3}) in some areas of the samples contains more than two maxima, which could not be conclusively associated with mismatch or misorientation in some regions. A comparison of this measurement with recently published results of rocking curve measurements on 4H-SiC epilayers suggests the presence of high density of

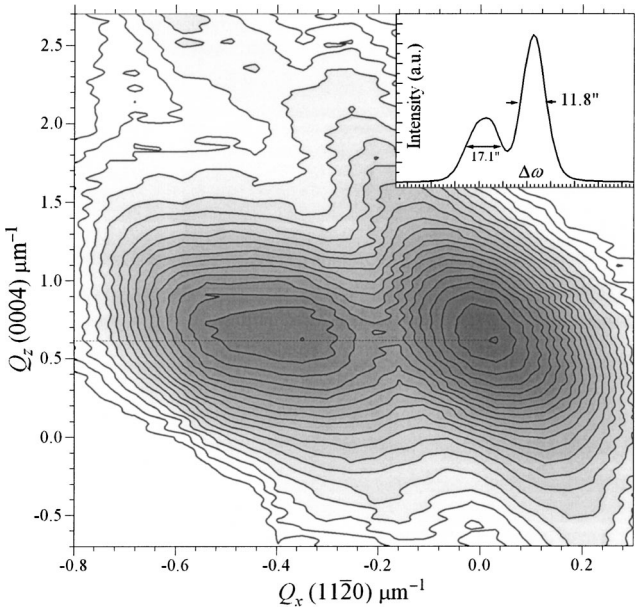

FIG. 4. A well-shaped double-peak RSM taken from a 4H-SiC substrate without an epilayer. The peak splitting is caused by small-angle grains in the crystal. Note that the *d*-spacing values of the two diffraction spots are the same. Inset is the corresponding double-axis RC with the FWHM values of the two peaks indicated.

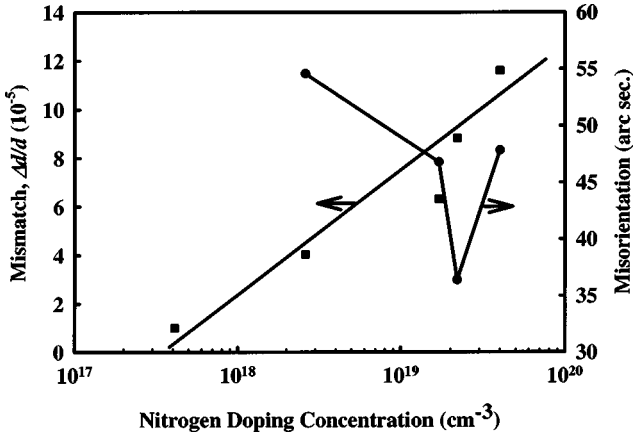

FIG. 5. Plot of spatially averaged lattice mismatch from the 0004 reflection in 4H-SiC epilayer as function of nitrogen doping level normalized. The 4×10^{17} cm^{-3} data point represents the measurement resolution. The lattice misorientations show no obvious dependency on the doping level.

domain boundaries in the two highly doped epilayer samples.[5–7] With regard to the observed large tilt of sample 66A [Fig. 1(b)], the corresponding SWBXT backreflection did not reveal any misfit dislocations that may be associated with relaxation due to tilt. Further, cross-section transmission electron microcopy did not show any misfit dislocations. Thus, if dislocations do exist, they must be in low density. If misorientation is in the substrate, it is usually caused by small-angle grains (or domains). Indeed, RCs of single crystal SiC frequently show multiple peaks. However, small-angle grains (as well as screw dislocations and micropipes) are related to lattice tilts (rotations) with no noticeable *d*-spacing change. Therefore, multiple diffraction spots arising from misorientations in the substrate should be exactly on a horizontal line, as shown in the RSM of Fig. 4, which is taken from a bare substrate. Therefore, we conclude that the tilt in the epilayer is likely due to the epilayer growth condition and not due to large tilts in the substrate.

As can be seen from Fig. 5, the magnitude of the misorientation between the epilayer and substrate has no obvious trend with respect to the doping level. However, detailed RSM measurements show that the lattice mismatch $\Delta d/d$ increases monotonically with increasing nitrogen dopant concentration.

In summary, measurements reported in this work have determined that in single crystal 4H-SiC, increasing the nitrogen doping level above 4×10^{17} cm^{-3} results in corresponding increase in lattice contraction. Also, significant lattice tilts exist, as revealed by the multiple subsidiary peaks in the HRXRD reflections, in epilayers doped greater than 2×10^{19} cm^{-3}.

[1] Cree, Inc., 2810 Meridian Parkway, Suite 176, Durham, NC 27713.
[2] Bede Scientific, Inc., 14 Inverness Drive East Suite H-100, Englewood, CO 80112.
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